IS 11720-6 (2001): Methods of Test for Synthetic Rubber, Part 6: Determination of Solvent Extract [PCD 13: Rubber and Rubber Products]

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IS 11720 (Part 6) : 2001

(Reaffirmed - 2012)

भारतीय मानक

संश्लिष्ट रबड़ की परीक्षण विधियाँ

भाग 6 विलायक निष्कर्षण ज्ञात करना

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 6 DETERMINATION OF SOLVENT EXTRACT

ICS 71.040.40:83.060

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

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Price Group 3

FOREWORD

This Indian Standard (Part 6) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

The concerned Committee has decided to prepare common methods of test for synthetic rubber under SR (Synthetic Rubber) series, namely, IS 11720 and this will be applicable to all types of synthetic rubbers being produced indigeneously. This standard (Part 6) is the sixth in the series. The other standards of this series are as follows:

Part 1 Methods of test for synthetic rubber : Part 1 Determination of antioxidants (SR:1)

Part 2 Methods of test for synthetic rubber: Part 2 Measurement of vulcanization characteristics with oscillating disc curemeter (SR:2)

Part 3 Methods of test for synthetic rubber: Part 3 Determination of mooney viscosity

Part 4 Methods of test for synthetic rubber: Part 4 Determination of volatile matter

Part 5 Methods of test for synthetic rubber: Part 5 Determination of ash

Part 11 Methods of test for synthetic rubber: Part 11 Rubber raw styrene- butadiene — Determination of soap and organic-acid content

Part 13 Methods of test for synthetic rubber: Part 13 Determination of Gel content

In the preparation of this standard, considerable assistance has been derived from ISO/DIS 1407 Rubber — Determination of solvent extract issued by the International Organization for Standardization (ISO).

The composition of the Committee responsible for formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if final value, observed or calculated is to be rounded off, it shall be done in accordance with IS 2 : 1960 `Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF TEST FOR SYNTHETIC RUBBER

PART 6 DETERMINATION OF SOLVENT EXTRACT

1 SCOPE

1.1 This Indian Standard (Part 6) prescribes the quantitative determination of total extractable materials from raw rubbers both natural and synthetic (SBR, CR, NBR, IR and IIR) and their unvulcanized and vulcanized compounds.

1.2 Two methods are adopted here:

a) Method A is a rapid extraction method; and
b) Method B is for use in case of dispute.

1.3 These methods are intended to determine the various organic constituents in the rubber like rosin, fatty acids, soaps extender oils, defoamer tars, antioxidants and other uncombined organic constituents which are extractable in the solvent used. The rubber hydrocarbon can also be estimated by substracting the sum of the total extract, the total ash and the total volatile matter from 100.

2 REFERENCE

The following Indian Standard contains provisions which, through reference in this text, constitute provisions of this standard. At the time of publication the edition indicated was valid. All standards are subject to revision, and parties to agreements based on the standard is encouraged to investigate the possibility of applying the most recent edition of the standard indicated below:

IS No.	*Title*
4518 (Part 1) : 1967	Methods of test for styrene-butadiene rubbers (SBR): Part 1 Determination of volatile matter, total ash, organic acid, soap, antioxidants, bound styrene and moony viscosity

3 LIMITATIONS

3.1 These methods are applicable to the rubbers given in Table 1.

3.2 The solvents recommended for individual rubbers are listed in Table 1, although other combinations may be desirable in certain cases. For instance, alcohols are particularly useful for extraction of vulcanized IR, NR and SBR where the extract will be used for thin-layer chromatography and not primarily for quantitative determination of amount of extractables.

3.3 The same quantitative results may not necessarily be obtained with either Method A or Method B not with the different solvents.

Method A generally gives results which are lower than those obtained with Method B due to an equilibrium being set up particularly if large test portions are used.

Table 1 Recommended Solvents
(*Clause* 3.1)

Sl No.	Rubber[1)]	Raw Rubbers and Unvulcanized Compounds	Vulcanizates
(1)	(2)	(3)	(4)
i)	IR, NR	Acetone[2)]	Acetone[2)]
ii)	SBR	ETA	ETA
iii)	CR	Propan-2-ol	Methanol
iv)	NBR	Propan-2-ol	Propan-2-ol
v)	IIR	Acetone[2)]	Acetone[2)]

[1)]IR - Isoprene Rubber, NR - Natural Rubber, SBR - Styrene-Butadiene Rubber, CR-Chloroprene Rubber, NBR - Acrylonitrile Butadiene Rubber and IIR - Isobutene-Isoprene Rubber

[2)]When acetone is used it may be partially converted into high boiling diacetone alcohol. If a difficult evaporation of the solvent is observed during the drying step, due to the presence of diacetone, repeat the extraction using a different solvent or use Method B.

4 REAGENTS

WARNING: All recognized health and safety precautions should be taken when using the reagents.

During the analysis, use only solvents recognized analytical grade.

4.1 Acetone

4.2 Ethanol toluene are azeotropic mixture (ETA), a mixture of 70 volumes of ethanol and 30 volumes of toluene. Reflex for 4 h over freshly calcined calcium oxide. Distill and collect a middle fraction with a boiling range of not more than 1°C. If absolute ethanol is used, the drying over calcium oxide may be omitted.

4.3 Propan-2-ol (Iso-propanol)

4.4 Methanol

5 APPARATUS

5.1 Extraction Apparatus

The following three types of extraction apparatus are described. Any other type of apparatus which performs the same function may be used:

a) The Type 1 apparatus comprises a receiver flask, jacketed soxhlet extractor and a condenser as shown in Fig. 1.

b) The Type 2 apparatus comprises a 250 ml receiver flask, a condenser and an extraction cup suspended by clean wire as shown in Fig. 2.

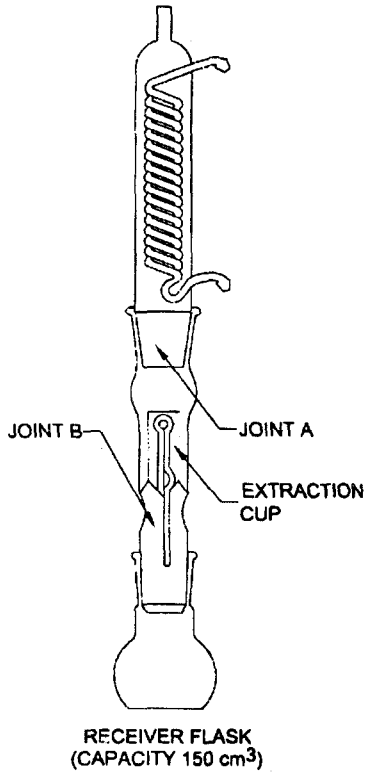
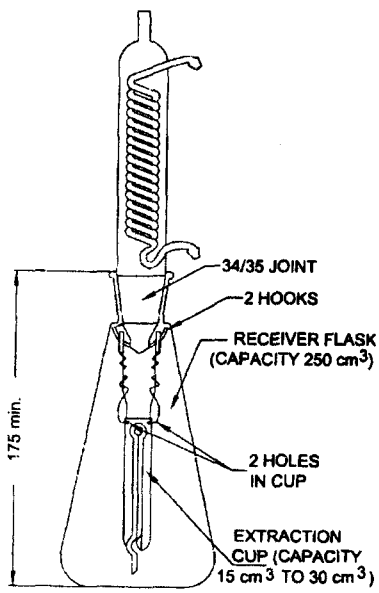


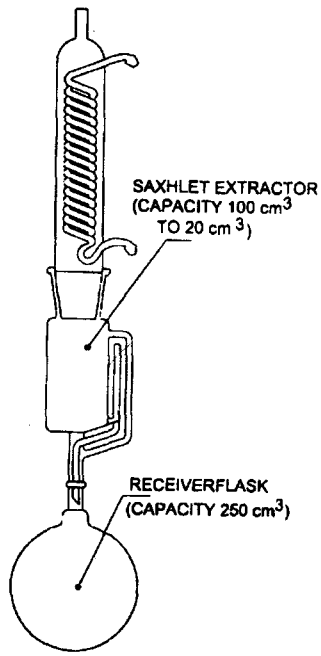
FIG. 1 TYPE 1 ALL-GLASS EXTRACTION APPARATUS



All Dimensions in millimetres.

FIG. 2 TYPE 2 ALL-GLASS EXTRACTION APPARATS

c) The Type 3 apparatus comprises a 250 ml receiver flask, a condenser and a Soxhlet extractor with a side arm as shown in Fig. 3. Typical Soxhlet capacity is 100 ml to 200 ml. There is no extraction cup.



All Dimensions in millimetres.

FIG. 3 TYPE 3 ALL-GLASS EXTRACTION APPARATUS

5.2 Distillation head and condenser (not shown in the figures), **appropriate for the apparatus** to distill of the **solvent after extraction.**

5.3 Oven operating at 100 ± 2°C.

5.4 Filter paper or nylon filter cloth, extracted before use with the same solvent as that used for the extraction.

5.5 Hot Plate

5.6 Sieve 150 Micrometer (100 Mesh)

5.7 Method for Taking Out Samples

The method for taking out samples shall be in accordance with the method prescribed in 3 of IS 4518 (Part 1) for raw styrene-butadiene rubbers (SBR). Take a portion of the raw rubber at least 250 g collected as above and pass it through the cold rolls of a laboratory mill set to a nip not exceeding 0.5 mm. For vulcanizates, the analyst shall, by inspection, assure himself that the vulcanizates has not been contaminated. The sample to be analysed shall be selected by taking pieces for various parts of the original sample and separating them from foreign matter. For soft vulcanized rubber it is preferable to mix the sample and grind it by passing it two or three times through a clean, cold, laboratory rubber-mill. The rubber will come from the mill in the form of a coarse powder or a rough sheet. If the product

is in the form of a sheet, the adjustment of the mill shall be such that the thickness of the final sheet is not greater than 0.5 mm. If the milled sample is a powder, it shall be transferred to a No. 14 (1.40 micrometer) sieve and rubbed through the sieve. Grinding shall be continued until the entire sample passes through the sieve. In the absence of milling machinery, the sample may be prepared by cutting it with scissors so that it will pass a No. 14 (1.40 micrometer) sieve and the cutting shall be continued until the entire sample passes through the sieve.

6 METHOD A

6.1 Outline of the Method

6.1.1 A weighed test portion of the rubber is extracted with ethanol toluene azeotrope solvent in a suitable apparatus (*see* Fig. 1 to 3). The solvent is distilled off and the residue is dried and weighed.

6.1.2 Take duplicate test portions from the sample prepared in accordance with **4.7**. Take a test portion of about 3 g to 5 g and weigh it to the nearest 0.1 mg (mass M_1).

6.1.3 Dry the chosen flask in the oven at 100 ± 2°C. Remove the flask from the oven and allow it to cool to room temperature in a desiccator, weigh to the nearest 0.1 mg (mass M_2).

6.1.4 Roll the weighed test portion in filter paper or nylon filter cloth to form a loose roll from which the rubber cannot fall and so that no part of the rubber is anywhere in contact with any other part of the rubber. If the test portion is in the form of small pieces, make a loose pocket of pieces in filter paper or nylon filter paper or nylon filter cloth. Fasten each test packet with clean wire. Place the packet in the appropriate extraction apparatus (*see* Fig. 1 to 3). Sufficient appropriate solvent to fill the extraction cup two or three times and pour into the extraction flask adjust the heating rate so that the quantity of distilled solvent fills the extraction cap 10 to 20 times per hour and extract for 16 ± 0.5 h.

6.1.5 At the end of the heating period, turn off the heating device, allow the apparatus to cool, remove the extractor or siphon cup and discard the rubber test portion unless it is required for further testing.

6.1.6 Dry the flask and contents for 2 h at 100 ± 2°C in the oven and, at the end of this time, remove the flask from the oven, cool in a desiccator and weigh to the nearest 0.1 mg (mass M_3).

6.1.7 Carry a blank through the entire procedure, using the same type of apparatus and quantity of solvent as for the test portion, but omitting the test portion, (Increase in mass, M_4).

6.2 Calculation

The solvent extractable material expressed as a percentage by mass, is given by the formula:

$$\frac{M_3 - M_2 - M_4}{M_1} \times 100$$

where

M_1 = mass, in grams of the test portion (*see* **6.1.2**);

M_2 = the mass in grams, of the empty receiver flask (*see* **6.1.3**);

M_3 = the mass, in grams of the receiver flask plus the extract after drying (*see* **6.1.6**); and

M_4 = increase in mass, in grams, of a receiver flask during the blank test (*see* **6.1.7**).

6.3 Report

6.3.1 The report shall include the following:

a) Proper identification of sample and reference standard;

b) Sample preparation;

c) Type of extraction apparatus; and

d) Result obtained.

NOTE — The results obtained may be different depending on the method of sample preparation.

7 METHOD B

7.1 Cut test portions from sample prepared as in **4.8** weighing between 90 mg and 110 mg nearest to 0.01 mg. Take 4 to 6 test portions (mass M_1).

7.2 Assemble the extraction apparatus (*see* Fig. 1 to 3) but cannot only the condenser to the flask No. Soxhlet, extractor cup or siphon cup is necessary - only the receiving flask and the condenser. The test portion is placed directly in the solvent. Alternatively, any arrangement for boiling solvent and test portions can be used, for example a beaker covered with a watch glass, a beaker covered with another beaker containing cold water, etc. The solvent volume shall be held constant and may be replenished with fresh solvent should evaporation occur.

7.3 Add about 25 ml of solvent for each test portion. Use a maximum of six test portions in a 250 ml receiver flask and four test portions in a 150 ml flask. Reflux for 60 min.

7.4 At the end of the heating period, turn off the heating device, allow the apparatus to cool, then remove the receiver flask from the condenser. Pour the contents of the flask into a clean 150 micrometer sieve to recover the extracted test pieces. Discard the solvent in an appropriate manner.

7.5 Gently bolt the extracted test portions between absorbent tissue to remove excess solvent and dry them separately at 100±2°C for 30 min or until the mass does not change by more than ±0.1 mg over a period of 10 min.

7.6 Cool the test portions for about 10 min in a desiccator and weigh each to the nearest 0.1 mg (mass M_2).

7.7 Calculation

The solvent-extractable material, expressed as a percentage by mass, is given by formula:

$$\frac{M_2 - M_1}{M_1} \times 100$$

where

M_1 = mass, in grams, of the test portion before extraction (*see* **7.1**); and

M_2 = mass, in grams, of the test portion after extraction (*see* **7.6**).

7.8 Test Result

A test result is the average of two determinations.

7.9 Test Report

The test report shall include the following:

a) a reference to this standard;
b) full identification of the rubber tested;
c) the method of sample preparation;
d) whether method A or method B was used;
e) which solvent was used;
f) which apparatus was used for extraction; and
g) the mean of the two determinations.

ANNEX A

(Foreword)

COMMITTEE COMPOSITION

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